DOCUMENT RESUME

ED 129 621 SE 021 485

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TITLE Comprehensive Qualitative Analysis for High School Chemistry.
PUB DATE [76]
NOTE 13p.; 1976 Science Teaching Achievement Recognition (STAR) award winning paper; Not available in hard copy due to marginal legibility of original document

EDRS PRICE MF-$0.83 Plus Postage. HC Not Available from EDRS.
DESCRIPTORS *Chemical Analysis; *Chemistry Instruction; *Laboratory Procedures; Laboratory Techniques; *Science Education; Secondary Education; *Secondary School Science

ABSTRACT

Described is a qualitative analysis laboratory procedure for high school chemistry students. The paper received the Science Teaching Achievement Recognition (STAR) award. The procedure acquaints students with certain ions and uses the knowledge of ions to devise a flow-chart for separating and identifying these ions in an unknown solution. (SL)

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COMPREHENSIVE QUALITATIVE ANALYSIS FOR HIGH SCHOOL CHEMISTRY

by Eyrl Short

Introduction

Qualitative analysis has been a part of college chemistry courses for many years. The chemical literature contains a variety of analytical schemes which can be used for a whole or part of a course in qualitative analysis.

High school chemistry students generally have been introduced to qualitative analysis through the flame tests, tests for the Halogens, and other selected tests for elements or ions. These introductory tests have generally been performed in isolation from each other and not placed together in a qualitative analysis scheme (3,4,5,8). Some procedures have been suggested that either, (1) require the student to use analysis schemes that have been constructed for the student, (2) analyze for only + ions, or (3) use a new sample of the unknown for each sub-group analysis (1,2,6).

This article describes a qualitative analysis procedure that has been used successfully by beginning high school chemistry students. The procedure is more cohesive than the isolated tests generally used in high school courses but less detailed than those in most college courses. The procedure involves the following 3 phases:

1. The students are given 8 metal ions and 5 non-metal ions. They experimentally determine how these ions react with or do not react with some selected tests reagents.

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2. The students then use the data they have collected to design a flow chart that will enable them to separate and identify the presence of the metal ions. The students also prepare a flow chart for the non-metal ions analysis.
3. Using the prepared flow charts, the students analyze an unknown solution.

Advantages

In this day of technological advancement, many high school students perceive the analysis of an unknown material to involve 'feeding' some information into a machine and obtaining an answer. Most students lack the understanding of the amount and kind of preliminary experimentation necessary if one is to be able to do this type of analysis. This procedure provides the students with such insight. This is further emphasized by having the students consider the amount of experimentation necessary if the teacher had not given them the selected test reagents.

Another advantage to this procedure is that it requires the student to examine a body of data and then devise a scheme to analyze an unknown. The student discovers that the non-reaction between an ion and a reagent may sometimes be as important as if there is a reaction.

This procedure requires the student to consider precipitation and separation of ions as part of the analytical process. The student learns that it is necessary to test for complete removal of certain ions before attempting confirmatory tests for the presence of other ions.

Preliminary Laboratory Procedures

As an introduction to this lab work, a demonstration lab is done using four sample solutions and three test solutions. By inductive discussion the students are led to suggest the need for compiling a body of knowledge showing how each of the four sample solutions react with the three test solutions. Many students are able to use this completed data table to suggest a possible flow chart for separation and identification of any combination of the four sample solutions. This suggested flow chart is then used to analyze an unknown composed of some combination of the four sample solutions. This demonstration lab is done using 13 X 100 mm. test tubes with approximately 1 ml. portions of the samples and small amounts (drops) of the test solutions, just as the students will use for their lab work. The demonstration also provides for the discussion of the use of the centifuge and other techniques such as decanting, testing for completeness of precipitation after decanting, and the use of a flow chart.

The students are given the instruction sheets, as shown in Appendix A, asked to make written preparation of the procedures, and to establish blank data tables prior to their next lab period. For these preliminary tests the students are allowed to work individually or with a lab partner. It requires approximately six 42-minute lab periods for most students to do these preliminary tests for the eight positive ions and five negative ions, as well as the special tests described under Item III on the instruction sheets.

When the preliminary tests have been completed, the student is told to devise a flow chart using these test results and the 'Ground Rules', as shown under Item IV on the instruction sheets.

The development of the flow chart has been done by requiring each student to individually present to the instructor a possible flow chart and then defend

this procedure and answer questions about the flow chart. If the student has overlooked some of the tests or arranged them improperly, the student is asked to attempt to correct the procedure and then return to the instructor for more discussion.

An alternate method for development of the flow chart has been to use class discussion as a means of arriving at an acceptable flow chart.

For the ions being used in this lab, there are not too many alternate methods of analysis. The most commonly suggested flow charts are shown in Appendix B and Appendix C.

In order to test the student's understanding of the flow charts and provide some practice in the use of the flow charts, several test tube racks containing test results for simulated unknowns are prepared and given to the students for practice. The student may do as many of these simulated unknowns as necessary for understanding and confidence.

Analysis of the Unknown

Each student is required to individually analyze an unknown solution containing from one to four ions. Each student is given a numbered bottle containing approximately 15 ml. of solution. While in the lab, the student is not allowed to discuss the analysis with anyone except the instructor. Obviously the students will discuss their work outside the lab and this is encouraged as a means of assisting those students who may be interpreting tests incorrectly or using the flow charts incorrectly. The student continues to analyze and submit answers until the correct ion or combination of ions is obtained.

Approximately 40 different unknowns have been prepared for analysis.

This number of unknowns provides an adequate variety for approximately 250 Chemistry students.

There has been a very good percentage of students being able to successfully analyze their unknown and obtain the correct answer the first or second time. This has been true for students of all levels of ability. For two consecutive years (1970-71 and 1971-72) a measure of the success of obtaining the correct answer was calculated for each of the 40 unknowns by dividing the number of students that analyzed a particular unknown into the number of times it took the students to obtain the correct answer. For example, if six students had an unknown and three students had it correct the first time and three had it correct the second time, the measure of success was obtained by dividing 6 into 9 to obtain 1.5. The calculated values for the measure of success ranged from 1.1 to 1.9, with 29 of the values being from 1.4 to 1.7.

The student interest in this lab has been very high. This interest has not been measured quantitatively but the best illustration of this interest is reflected by students from years past asking if the unknown analysis is still being done in the Chemistry course.

Course Applications

This unknown lab has been used from approximately the twelfth to the nineteenth weeks of the course. This places the lab early enough in the course so there can be frequent reference to this work for the remaining twenty weeks of the course. There are many points of application: relative reactivity of the halogens, spectra, solubility of solids and liquids, equilibria, acid-base reactions, as well as many descriptive chemical properties of the substances used for this lab.

This lab also lends itself to being open-ended with the students sometimes getting unexpected results when analyzing their unknown. Illustrations of these kinds of results would be: the result of the Chlorox test when the unknown has Mn^{+2} present or the appearance of a small amount of PbS precipitate after the Pb^{+2} has apparently been removed by treating with excess HCl. This allows apportunity to challenge the high ability student to conduct further investigation into such problems.

Summary

This article has described a qualitative analysis laboratory procedure for high school Chemistry students that acquaints the students with a series of test to build a body of knowledge concerning certain ions and then uses this knowledge to devise a flow chart that will enable the student to separate and identify these ions in an unknown solution.

This lab has been used for a number of years in its present form. However, the procedure also lends itself to further modification and could easily incorporate the procedure for separating metallic ions by sequential precipitation as reported by Pearson (7).

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APPENDIX A

Our approach to qualitative analysis is (1) to acquaint you with a series of tests to build a body of knowledge concerning certain ions, (2) to use this body of knowledge to devise a "flow chart" scheme for identifying any of the ions, (3) to identify the ions in your unknown.

The tests suggested below should enable you to devise a scheme on your own and then to individually identify the 1-4 ion(s) in your "unknown". Each person may work at his own rate, and at times other than regularly scheduled lab periods. A final completion date will be set after about 2-3 weeks of work. The lab will last 5-6 weeks in total.

I - Carefully do each of the following tests. Make and record complete observations.

1a. Take a flame test wire and clean it by heating in the hottest part of a burner flame until an initially cool wire shows no flame color prior to the wire's becoming "red hot". When this is reached the wire is "clean". Note: a "red hot" wire vaporizes slightly, yielding a faint flame color.

b. Dip the wire into a sample (1 ml) of a Mn^{+2} solution and place in the flame. Note any new flame color prior to the wire's becoming "red-hot". Repeat several times until you can make a definite decision of any new flame color. Clean the wire and repeat the test successively with Ag^{+}, Pb^{+2}, Cd^{+2}, Zn^{+2}, Ba^{+2}, Sr^{+2}, Na^{+}.

2. In separate test tubes pour approximately 1 ml. of each of the 8 metal ion solutions. To each add 8-10 drops of TA (thioacetamide) solution. Add 6 drops 15 M NH_3 solution (sometimes labeled "15 M NH_4OH"). Place 1 drop of resulting mixed solution on indicator paper using a stirring rod. If paper turns blue, proceed to the next step; if not, repeat the addition of NH_3 and testing until blue indicator paper is achieved. Place in boiling water bath in the HOOD for 4-6 minutes. Observe, then discard the solution into the waste beaker in the HOOD.

3. In separate test tubes pour approximately 1 ml. of 8 metal ion solutions. Add 4-6 drops of HCl (hydrochloric acid) solution. Observe and discard the solutions into the waste beaker near the front sink.

II. Carefully do each of the following tests on each of the negative ions. Make and record complete observations.

1. In separate test tubes pour approximately 1 ml. of the following ion solutions: Cl^-, Br^-, I^-, CO_3^{-2}, SO_4^{-2}. To each add 8-10 drops of $AgNO_3$ (silver nitrate) solution.

2. In separate test tubes pour approximately 1 ml. of the negative ion solutions. Add 8-10 drops of barium nitrate, $Ba(NO_3)_2$.

III. Carefully do the following tests on the specific ion noted. Make and record complete observations.

1. In separate test tubes pour 1 ml. of Br^-, I^-, and Cl^- solutions and 1 ml. distilled water. To each add an equal volume of CCl_4. To each add 4-6 drops "chlorox" and 2-3 drops HCl. Cap the tube with thumb, shake carefully at first, release any pressure, then shake vigorously. Observe and discard into the waste beaker in front of room.

2. In separate test tubes pour approximately 1 ml. of CO_3^{-2} and SO_4^{-2} ion solutions. Add 8-10 drops of barium nitrate solution to each. Add 6-8 drops of HCl to each and shake cautiously, then more vigorously. Observe and discard.

3. In separate test tubes pour approximately 1 ml. of Ag^+ and Pb^{+2} ion solutions. Add 4-6 drops of HCl solution. Centrifuge and discard the liquid portion. Wash the ppt. with 1 ml. of distilled water and discard the wash water. Add another 1 ml. of distilled water, shake and place in a boiling water bath for 5-7 minutes. Pour off the liquid into another test tube and test it with 4-6 drops of K_2CrO_4. Observe and discard in the front waste beaker.

IV. Devise a flow-chart procedure which would allow you to identify any one of the 13 ions. It is suggested you analyze the positive ions with one sample and the negative ions with another sample.

Ground Rules:

1. Your unknown will contain 1-4 ions from the scheme. (It may contain others not in the scheme which you will not be expected to identify).

2. You will have no more than 1 from any of the following groups (you may have none from a particular group).

A. Cl^-, Br^-, I^-

B. SO_4^{-2}, CO_3^{-2}

C. Sr^{+2}, Ba^{+2}, Na^+

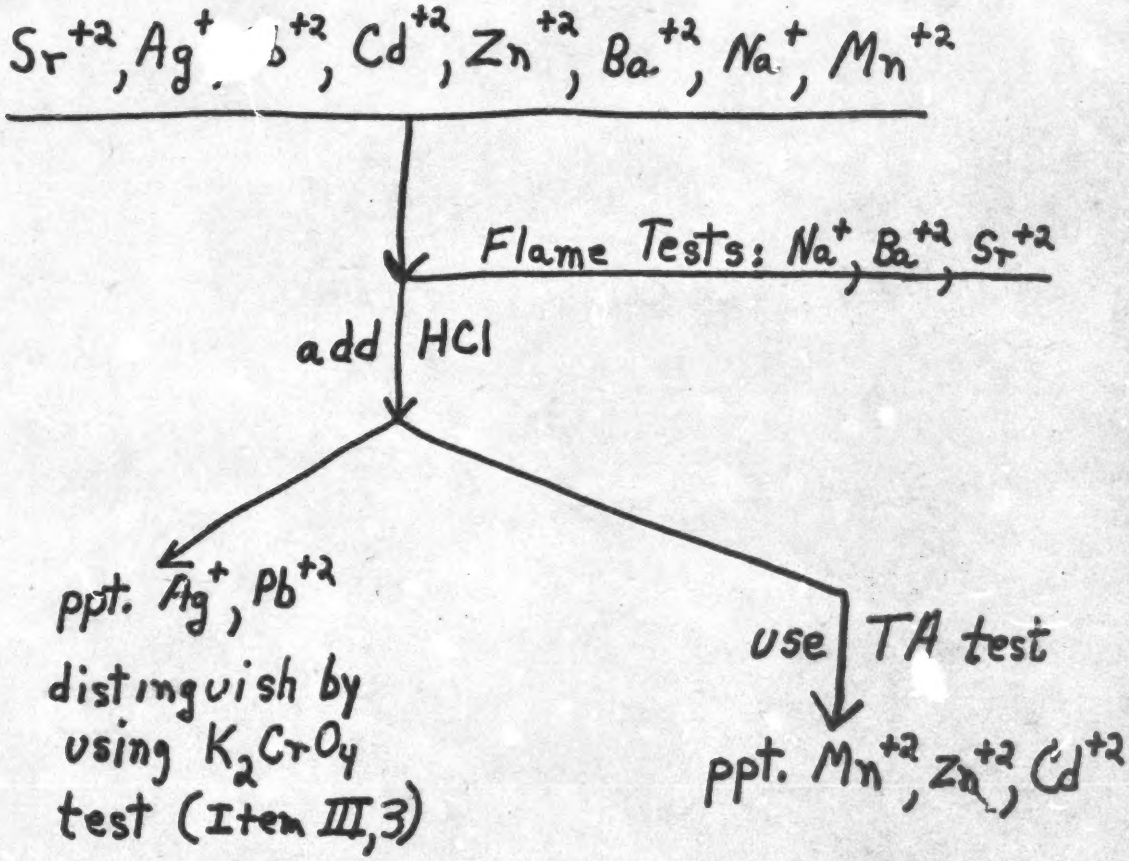
D. Ag^+, Pb^{+2}

E. Cd^{+2}, Zn^{+2}, Mn^{+2}

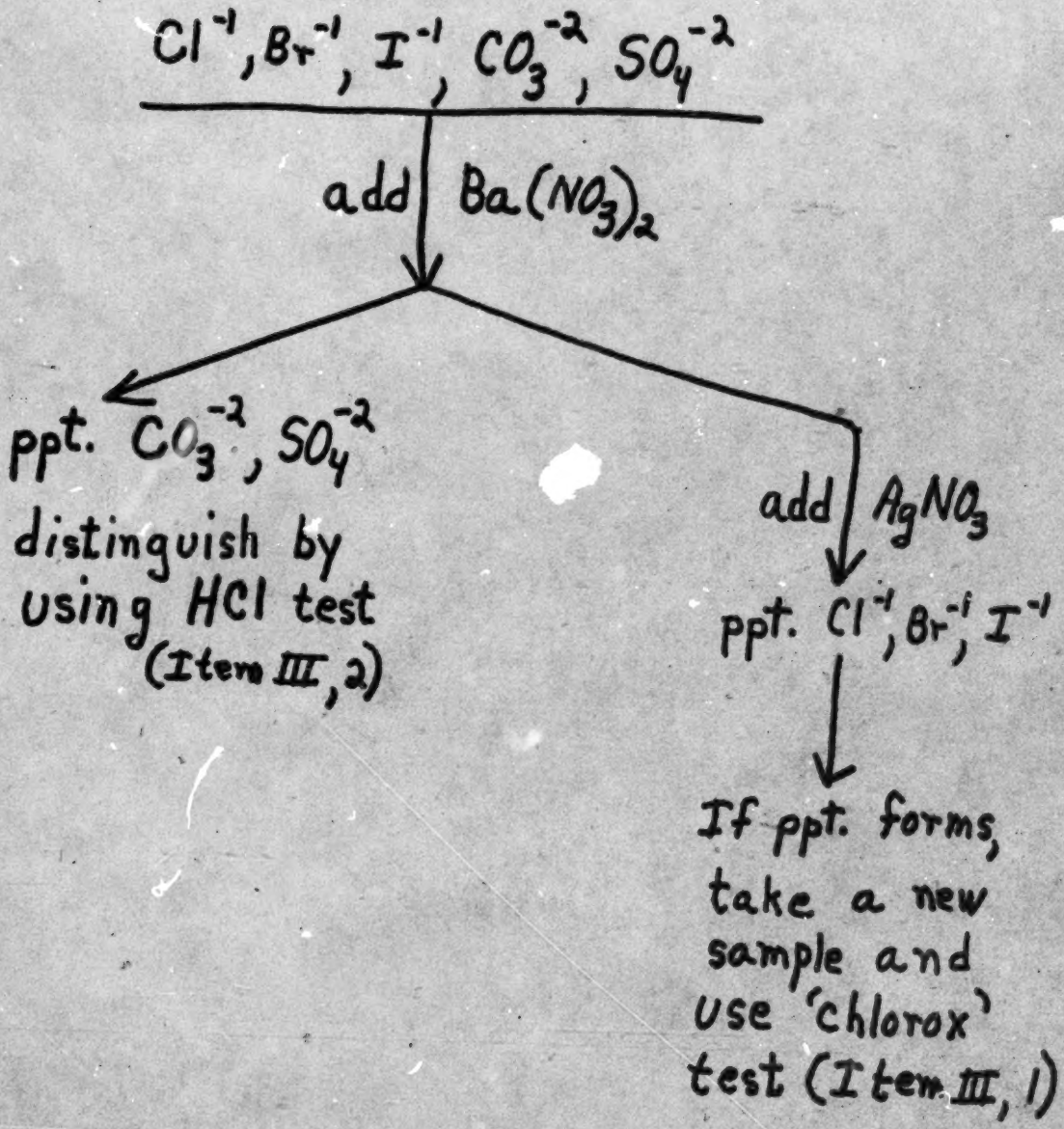
3. Each person is on his own and should confer only with the instructor.

V. Have your flow chart checked, receive and analyze your unknown. When you are satisfied with your analysis submit your name, unknown number and list of ions on a piece of paper to your instructor.

Appendix B


$Sr^{+2}, Ag^{+}, Pb^{+2}, Cd^{+2}, Zn^{+2}, Ba^{+2}, Na^{+}, Mn^{+2}$
Flame Tests: Na^{+}, Ba^{+2}, Sr^{+2}
add HCl
ppt. Ag^{+}, Pb^{+2}
distinguish by using K_2CrO_4 test (Item III,3)
use TA test
ppt. Mn^{+2}, Zn^{+2}, Cd^{+2}

Appendix C


$Cl^{-1}, Br^{-1}, I^{-1}, CO_3^{-2}, SO_4^{-2}$
add $Ba(NO_3)_2$
ppt. CO_3^{-2}, SO_4^{-2}
distinguish by using HCl test (Item III, 2)
add $AgNO_3$
ppt. Cl^{-1}, Br^{-1}, I^{-1}
If ppt. forms, take a new sample and use 'chlorox' test (Item III, 1)